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Standard Method of Test for
TOTAL CHLORINE IN VINYL CHLORIDE POLYMERS AND COPOLYMERS[1]

This Standard is issued under the fixed designation D 1303; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the determination of total chlorine in vinyl chloride polymers and copolymers. The method does not distinguish between poly(vinyl chloride) and poly(vinylidene chloride).

2. Summary of Method

2.1 The organic matter is destroyed by hot sodium peroxide, and the chlorine is converted to chloride. The chloride is precipitated as silver chloride with an excess of standard silver nitrate, and this excess is determined by titration with standard thiocyanate solution using ferric nitrate indicator and nitrobenzene to depress the solubility of the silver chloride. The amount of silver nitrate required to precipitate the chloride is a measure of the total chlorine content of the sample.

3. Apparatus

3.1 *Parr Peroxide Bomb*, Series 2100, 22-ml, gas-fired.[2]

4. Purity of Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals or equivalent, as specified in ASTM Methods E 200, Preparation, Standardization, and Storage of Standard Solutions for Chemical Analysis,[3] shall be used in all tests.

4.2 *Purity of Water*—Unless otherwise indicated references to water shall be understood to mean distilled water or water of equal purity.

5. Reagents and Materials

5.1 *Powdered Sugar* (preferably cp sucrose).

5.2 *Sodium Peroxide*, cp, calorific grade, assay 95 percent, min, as Na_2O_2 (Note 1).

5.3 *Nitric Acid* (*sp gr 1.42*)—Concentrated nitric acid (HNO_3).

5.4 *Silver Nitrate Solution* (*0.1 N*)—Prepare and standardize a 0.1 *N* solution of silver nitrate ($AgNO_3$).

5.5 *Ammonium or Potassium Thiocyanate Solution* (*0.1 N*)—Prepare and standardize a 0.1 *N* solution of either potassium thiocyanate (KCNS) or ammonium thiocyanate (NH_4CNS).

5.6 *Ferric Nitrate Solution* (*50 g* $Fe(NO_3)_3$ */liter*)—Dissolve 50 g of Ferric nitrate ($Fe(NO_3)_3$) in water and dilute to 1 liter.

5.7 *Nitrobenzene*, cp.

NOTE 1—Keep Na_2O_2 in a tightly closed container, and away from moisture.

6. Preparation of Sample

6.1 Dry the sample for 45 min in an oven at 100 ± 1 C, in an atmosphere of CO_2 or nitrogen. Store in a desiccator.

NOTE 2—Moisture pick-up is a problem, so the dried sample should be used promptly, and should not be stored for prolonged periods.

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at 23 ± 2 C (73.4 ± 3.6 F) and 50 ± 5 percent relative humidity for not less than 40 h prior to test in accordance with Procedure A of ASTM Methods D 618, Conditioning Plastics and Electrical Insulating Materials for Testing,[4] for those tests where conditioning is required. In cases of disagreement, the tolerances shall be 1 C (1.8 F) and ±2 percent relative humidity.

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[1] This method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D-20.70 on Analytical Methods.

Current edition effective Sept. 12, 1955. Originally issued 1953. Replaces D 1303 - 55 (1961).

[2] The Parr Peroxide Bomb may be obtained from the Parr Instrument Co., Moline, Ill. Parr electrically fired bombs may be used in place of the gas fired peroxide bombs, provided tests of both apparatus have been conducted to demonstrate reproducible agreement of results.

[3] *Annual Book of ASTM Standards*, Part 30.

7.2 *Test Conditions*—Conduct tests in the Standard Laboratory Atmosphere of 23 ± 2 C (73.4 ± 3.6 F) and 50 ± 5 percent relative humidity, unless otherwise specified in the test methods or in this specification. In cases of disagreements, the tolerances shall be 1 C (1.8 F) and ±2 percent relative humidity.

8. Procedure

8.1 To a clean, dry, 22-ml Parr bomb cup add 0.200 to 0.225 g of finely powdered sample. Add 0.5 g of dry powdered sugar and a standard measure (15 g) of Na_2O_2. Close the bomb tightly with the lid and the retaining rings, and shake vigorously to mix the contents. Tap the bomb lightly on the bench to dislodge particles sticking to the cap and to the upper portion of the bomb cup. Set the bomb over the hole in a steel housing supported on a tripod. A small steel chamber with an access door and a small sight hole built over the tripod is desirable for reasons of safety.

8.2 Add 2 ml of water to the depression in the bomb cover. Apply a very hot bunsen flame to the bottom of the bomb. Adjust the air to the burner to give a cone about 38 mm (1 $^1/_2$ in.) high, the apex of which should be just short of contact with the bottom of the bomb. Allow to heat until the water in the lid begins to boil or until a dark red ring appears on the side of the cup, then quench in cold water. **Caution:** The heating of the bomb is of extreme importance, since underheating will cause imcomplete oxidation of the organic matter and low chlorine results, while overheating will cause possible damage to the bomb cup and even an explosion. These bombs are not intended to operate at a red heat, but there is no serious danger involved in this method of oxidation provided the proper charges are used in a bomb that is in a satisfactory condition, and provided the proper heating technique is used (Note 3). Discard the 22-ml bomb cups if the sides or bottom become visibly swollen, or if the interior surfaces become worn or corroded to an inside diameter of 27.4 mm (1.08 in.) at any point.

8.3 When the bomb is cold, remove it from the quenching bath, rinse if off with water, and open. Place the bomb cup on its side in a clean 600-ml beaker and add water until about half covered. Rinse off the bomb cover thoroughly with water, catching the rinsings in the same beaker. Cover the beaker with a watch glass, and warm slightly until the contents of the bomb are dissolved. Remove the bomb cup and rinse thoroughly with water, catching the rinsings in the same beaker. Transfer quantitatively to a 500-ml glass-stoppered flask, add 2 to 3 drops of phenolphthalein indicator solution, neutralize with HNO_3, using care to avoid loss by spattering and overheating, and add an excess of 5 ml of HNO_3 (Note 4). Add 2 ml of nitrobenzene (Note 5) and 50.0 ml of 0.1 *N* $AgNO_3$ solution, stopper, and shake until the precipitated AgCl becomes a spongy mass. Add 10 to 15 ml of $Fe(NO_3)_3$ indicator solution and titrate with 0.1 *N* thiocyanate solution to the first pink end point.

8.4 Run a blank determination at least every time a change in solutions or reagents is made, to ensure against high results caused by chlorides in the reagents.

NOTE 3—A careful reading of Parr Manual No. 121 on the method and precautions used in this method is suggested.

NOTE 4—When properly fired, the bomb leachings are free of black particles, and the acidified solution is substantially clear. If not, a new sample must be prepared and fired.

NOTE 5: **Caution**—Nitrobenzene is toxic, and care must be used to avoid contact with the skin.

9. Calculations

9.1 Calculate the chlorine and vinyl chloride content as follows:

Total chlorine, percent

$$= [(V_1N_1 - V_2N_2)\,0.03546/W] \times 100$$

Vinyl chloride, percent

$$= [(V_1N_1 - V_2N_2)\,0.06246/W] \times 100$$

where:

V_1 = milliliters of $AgNO_3$ solution added to

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[4] *Annual Book of ASTM Standards*, Part 27.

the sample,
N_1 = normality of the $AgNO_3$ solution,
V_2 = milliliters of KCNS solution required for titration,
N_2 = normality of the KCNS solution, and
W = grams of sample used.

10. Reproducibility

10.1 Duplicate determinations should check within 0.3 percent.